

ATTACHMENT A

CALIBRATION AND QUALITY CONTROL PROCEDURES

Calibration and Quality Control Procedures for Method 4042

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW4042	Total DDTs	Two-point calibration standards at 0.2 and 1.0 mg/kg	Prepare and analyze during sample preparation and analysis for each batch	Response of the standards should be inversely relational to concentration	Reanalyze batch
		Method Blank	1 per batch	Response greater than the 0.1 mg/kg standard response	Investigate possible source of problem. Take appropriate corrective action. Reanalyze batch.
		Duplicate preparation and analysis	1 per batch	Equivalent result (< 0.2 mg/kg; $>0.2 < 1$ mg/kg; or >1 mg/kg)	Identify potential source of problem and correct. If source is not apparent, reanalyze same sample and duplicate in following batch to verify heterogeneity.

Calibration and Quality Control Procedures for Method 8081A

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8081	Total DDTs	Five-point initial calibration	Prior to sample analysis and when CCV fails	Option 1: RSD for each analyte $\leq 20\%$ Option 2: Grand mean RSD $\leq 20\%$, with no individual analyte RSD $>30\%$ Option 3: Linear regression $-r \geq 0.995$ Option 4: Non-linear regression COD $r^2 \geq 0.990$ (6 points for 2 nd order, 7 points for 3 rd order)	Correct problem then repeat initial calibration.
		Second source standard (not required if calibration verification below is prepared with a second source of the standard)	Following initial calibration	% Difference from expected value $\leq 15\%$ for all analytes OR grand mean $\leq 15\%$ with no individual response factor greater than 20%	Correct problem, rerun second source standard. If that fails, repeat initial calibration.
		DDT and endrin breakdown check	Daily prior to analysis of samples	Degradation $< 15\%$	Correct problem, then repeat breakdown check.
		Calibration verification	<u>ICV</u> : At the beginning of an analysis sequence <u>CCV</u> : After every 10 field samples and at the end of the analysis sequence	Response factor for all analytes within $\pm 15\%$ of initial calibration response factor OR grand mean within 15% with no individual response factor greater than 25%	<u>ICV</u> : Correct problem, rerun ICV. If that fails, repeat initial calibration <u>CCV</u> : Correct problem, then repeat CCV and reanalyze all samples since last successful CCV or ICV

Calibration and Quality Control Procedures for Method 8081A

Analytical Method	Applicable Parameter	Quality Control Check	Minimum Frequency	Acceptance Criteria	Corrective Action
SW8081	Organochlorine Pesticides	Method Blank	1 per preparation batch	All analytes < ½ QL.	Investigate possible contamination source. Take appropriate corrective action. Reprep and reanalyze all samples processed with a contaminated blank, unless analyte is not detected in associated samples or present at greater than 10x blank concentration.
		Laboratory Control Sample	1 per preparation batch	Comparison recovery limits 60-130%(water), 50-150% (soil)	Correct problem, then reprep and reanalyze LCS and all samples in the associated preparatory batch for failed analytes.
		Matrix Spike and Matrix Spike Duplicate	1 MS/MSD per 20 project samples when identified on the Chain-of-Custody	Comparison recovery limits 60-130% (water), 50-150% (soil) and RPD <35% for soil samples RPD <20 % for water samples	Evaluate for supportable matrix effect. If no interference is evident reprep and reanalyze MS/MSD and all samples in the preparation batch once within the holding time. If still out report both sets of data.
		Surrogate spike	All field and quality control samples	Comparison recovery limits 60-130% (water), 50-150% (soil)	Evaluate for supportable matrix effect. If no interference is evident reprep and reanalyze affected sample(s).
		Confirmation of positive results (second column or second detector)	All detected results at or above the QL must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and secondary column RPD ≤ 40%	None – report as detected result if criteria is met. Use professional judgment to determine whether primary or secondary column concentration should be reported. Report as not detected at QL if criteria is not met.
		Quantitation limit standard (lowest concentration on initial calibration curve)	Verify at least once for every matrix and field effort	QLs established shall not exceed those in the Appendix B tables.	QLs that exceed established criteria shall be submitted to USACE Project Chemist for approval prior to analysis of any project samples.